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REPUBLIC
AVIATION CORPORATION

RAC 921-1
(RD-QPR-63-619)

ENGINEERING PROGRESS REPORT ON INVESTIGATION OF THE BASIC MECHANISM OF METAL FATIGUE

RAC 921-1

28 October 1963

Prepared under Naval Bureau of Weapons

Contract NOw 63-0704-c

Quarterly Report No. 1

1 June 1963 through 30 September 1963

This report applies to work
on contract: NOw 63-0704-c

REPUBLIC AVIATION CORPORATION
Farmingdale, L. I., N. Y.

ABSTRACT

The mechanism of metal fatigue is being investigated by studying dislocation dynamics in single crystals during stress cycling by means of ultrasonic attenuation measurements in the megacycle frequency range. A basic and applied approach are being explored. The basic investigation will be limited to the first cycle of stress-essentially a study of the Bauschinger effect. The applied portion will deal with an evaluation of similar measurements made continuously, cycling to fracture. All studies will be performed on pure, annealed single crystals of definite orientation in order that the attenuation measurements will be susceptible to interpretation in terms of dislocation damping theories.

In this First Quarterly Progress Report, a method for growing oriented copper single crystals is described, and techniques for electrochemical cutting and chemical polishing are discussed.

Self-aligning tension-compression grips were designed and fabricated to facilitate study of the deformation of the copper single crystals.

The major preparatory work prior to studies of the deformation of the copper single crystals has been accomplished.

During the next period, oriented single crystal specimens of high purity copper will be grown from oriented seeds. The specimens will then be electrochemically cut and chemically polished. Attenuation measurements will be made on oriented single crystals during cyclic deformation.

CONTENTS

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SECTION I

INTRODUCTION

The basic mechanism of metal fatigue is one of the most important unsolved problems, common to both solid-state physics and engineering. The necessity for research to understand and ultimately to solve this problem is self-evident and requires no further justification. The literature recounting empirical observations on fatigue is voluminous and dates back over a century. The majority of the past experimental work has been concerned with providing engineering data for mechanical design. This type of data shows fatigue as a hopelessly complex phenomenon depending on a large number of variables. Recently, there has been a concerted effort to explore the basic mechanism of fatigue [1, 2, 3, 4, 5]. It is now evident that final explanation of this phenomenon will have to be given in terms of the lattice defects which determine the mechanical behavior of a crystalline solid. Specifically, the mechanism of metal fatigue is a problem in the realm of dislocation physics. Stripped of all its cumbersome trappings, metal fatigue is the result of the movement and interaction of dislocations activated by a cyclic stress.

The purpose of this research program is to elucidate the basic mechanism of metal fatigue by studying dislocation dynamics in single crystals during stress cycling. The behavior of dislocations under alternating stress will be described by ultrasonic (megacycle) attenuation measurements. The megacycle frequency range will be used because it has been shown to be a very sensitive means for studying dislocation movements and the interaction of dislocations with point defects. The ultrasonic technique permits a continuous interpretation of observations during stress cycling. Conventional mechanical property evaluations and advanced microscopic techniques require stopping the stress cycling at various times to evaluate the mechanical or microstructural properties of a test specimen. The experimental technique to be used offers other unique

advantages. It develops a conventional S-N curve as well as an attenuation versus N curve. The technique bridges the gap between conventional S-N information and the newer type of scientific information to be generated.

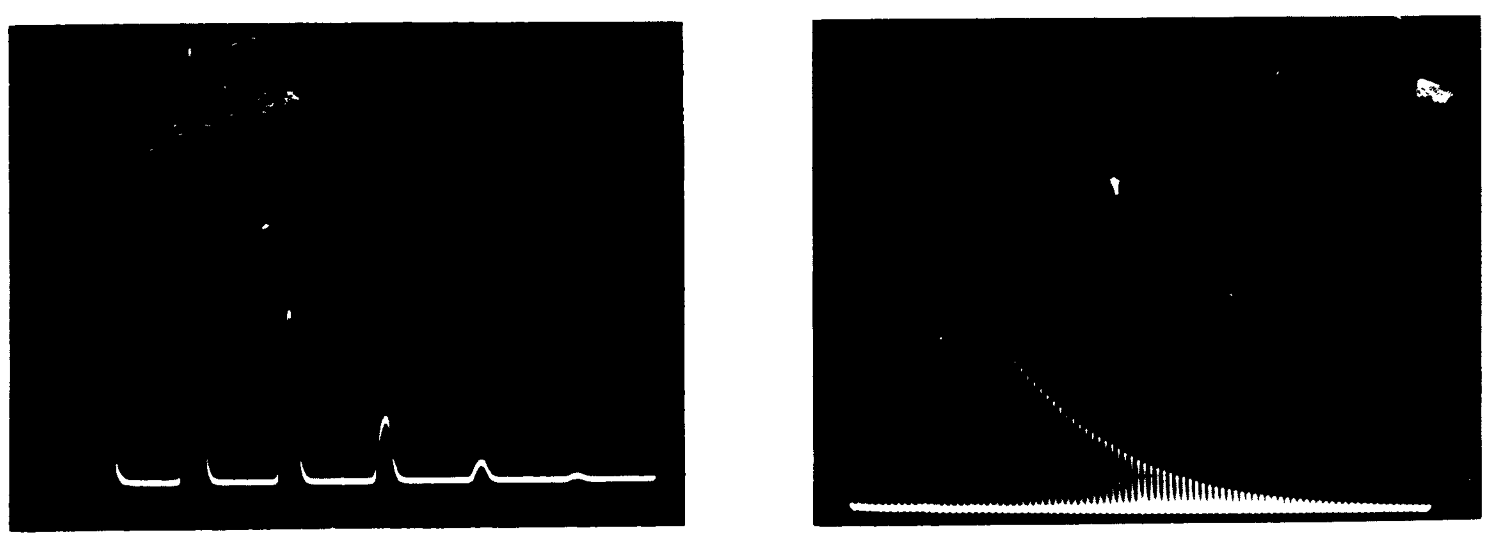
A basic and an applied approach will be explored. The basic investigation will be limited to an extensive study of the dislocation dynamics of the first cycle of stress - essentially a study of the Bauschinger effect. An understanding of the first cycle of the fatigue process appears to be a necessary first step in arriving at an explanation of fatigue. The applied portion will deal with an evaluation of similar measurements made continuously during cycling to fracture.

All studies are being performed on pure, annealed single crystals of definite orientations to minimize ambiguity in interpreting the damping measurements. The orientations chosen are pure mode axes, i.e., directions for which pure longitudinal and shear waves may be propagated independently.

It is necessary to avoid non-uniformities of stress which would lead to a spurious Bauschinger effect. Therefore, the experiments will be carried out under conditions of uniform strain (push-pull) to avoid macroscopic non-uniformity of stress such as is encountered in bending or torsion. The use of single crystals will avoid microscopic nonuniformities associated with polycrystalline specimens.

Apart from avoiding spurious Bauschinger effects, it is advisable to use single crystals in damping studies of this type because there is a large "background" loss in polycrystalline specimens due to grain boundary scattering. This is illustrated in Figure 1 where the echo patterns (at 30 mc/sec) for two tungsten specimens of the same length are shown; one a single crystal, the other a polycrystal. The marked damping in the polycrystal specimen is very striking. Furthermore, individual slip systems can be studied in single crystals because pure mode waves of a given polarization can be propagated along a definite orientation.

The face-centered cubic crystal structure system will be investigated. Copper and aluminum were considered as candidate materials because of



TUNGSTEN POLYXTAL TUNGSTEN SINGLE XTAL

Figure 1. Echo Patterns for Two Tungsten Specimens

their availability and the ease of growing them as single crystals. Copper was chosen because aluminum exhibits deformation recovery at room temperature[6].

The accomplishments to date include the development of techniques for growing, cutting, polishing, and testing copper single crystals. These are discussed in the following section.

SECTION II

ACCOMPLISHMENTS

A. GROWTH OF ORIENTED COPPER SINGLE CRYSTALS

A review was made of the existing methods of growing single crystals. It was decided to employ the Bridgman technique, utilizing a Lepel High Frequency Induction Power Supply (10 K.W.) and a Vertical Zone Refining Unit. The zone refiner (Figure 2) employs a helical coil attached to the power supply. This coil surrounds a quartz tube which encloses a graphite mold positioned on a stainless steel coupling.

The following procedure was used in growing the oriented single crystals. A 4-inch length of a seed crystal was loaded into a previously outgassed mold, and a known length of polycrystalline copper was placed on top of the seed. After sealing the assembly by placing the flanged head on the end of the quartz tube, the system was evacuated to 10^{-5} mm Hg. Next, the quartz-graphite assembly, which moves vertically, was positioned in the precalibrated helical induction coil, so that the top end of the single crystal seed and the adjacent polycrystal would melt when the power was applied. The assembly was then moved down through the coil at a rate of 1/4-inch per hour while a constant power was induced into the graphite-copper load. Vertical motion was automatically stopped when the end of the copper had traversed through the coil. Separate timers turned off the high and low voltage circuits.

Many problems were encountered and solved prior to the successful growth of oriented single crystals from seed crystals. Initially, several coil designs were tested in attempting to obtain both a narrow temperature gradient and sufficient power to melt. Those which did not produce the desired effect were a conical coil with a plate concentrator, a helical coil with close turns, and a helical coil with a concentrator on the bottom. The system that

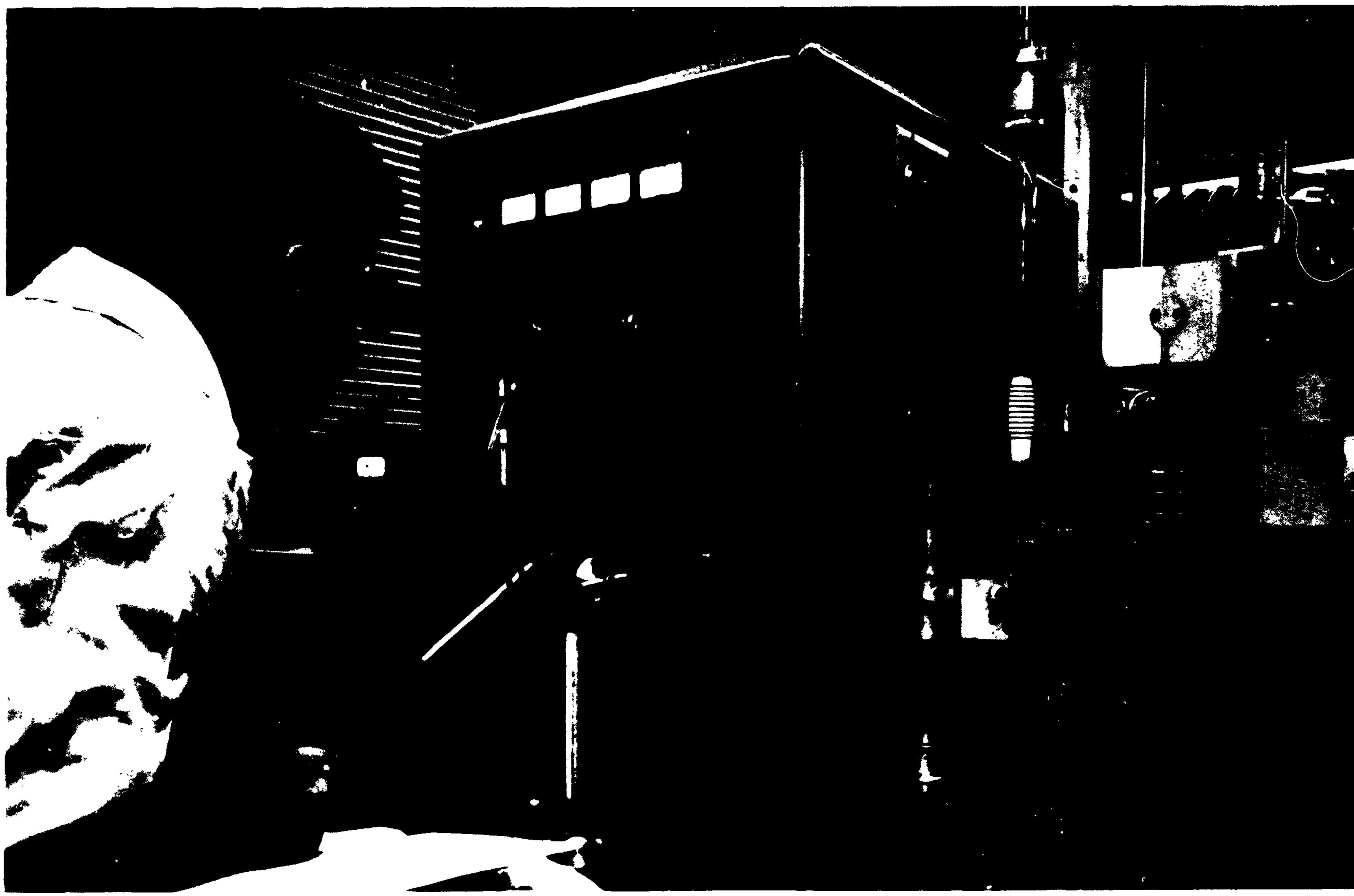


Figure 2. Lepel High Frequency Induction Power Supply with Vertical Zone Refining Unit Used for Growing Copper Single Crystals

proved to be satisfactory was a helical coil with a split copper disc concentrator at the center turn. The temperature gradients as well as the power settings required for melting were carefully established for several different lengths of polycrystalline copper. Temperature measurements made with a micro-optical pyrometer (disappearing type tungsten filament) were not accurate at the melting point of copper after prolonged periods of melting, due to vaporization and deposition on the quartz tube. The readings taken on the graphite, indicative of initial melting of a 10-inch copper length, were determined to be 1130°C. However, by allowing for absorption losses in the quartz tube and applying an emissivity correction for the graphite, the true temperature was calculated to be 1170°C, about 87°C above the melting point of copper. Subsequent increases in copper length above 10 inches necessitated an increase in power to achieve melting.

It was established experimentally that for a particular power setting, the grid and plate current values remained constant after many hours of melting, assuring very little temperature variation. Overheating produced two deleterious effects: excessive copper deposition on top of the graphite mold and the quartz tube, and accelerated diffusion of copper in the graphite to promote leakage. In the early experiments, National Carbon coarse-grained AUC graphite molds were found to be too porous, and leakage was common at normal operating temperatures. Fine-grained ATJ graphite did not leak and gave a superior quality machined internal surface*.

In the first attempts to grow single crystals from randomly oriented single crystal seeds of O. F. H. C. copper, only coarse polycrystalline lengths were produced. It was determined that a single crystal could be produced from a seed once a new gear reduction system was installed in the vertical zone refining unit to reduce the minimum rate of travel, from 3-inches per hour to less than 1/4-inch per hour.

The effect of growth rate on the substructure was determined. A single crystal was propagated into a length of 99.999 polycrystalline copper using growth rates of 1/4-inch per hour, 1/2-inch per hour, and 1-inch per hour

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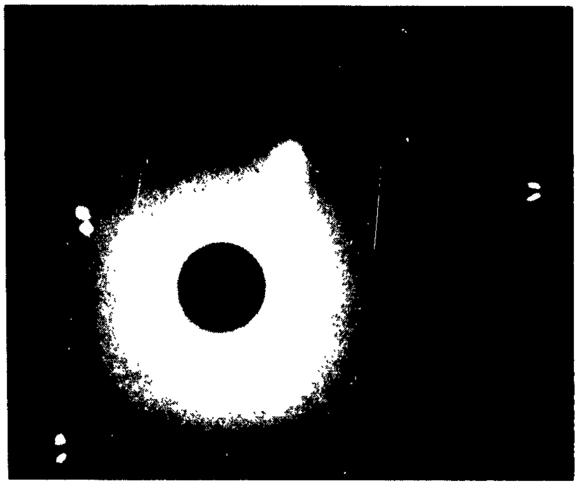
* Special reamers were ground to produce extremely smooth surfaces free of tool markings.

from an O. F. H. C. randomly oriented single crystal seed grown at 1/2-inch per hour. The results were similar to those of McGrath and Craig [7], who found that for 99.999% pure aluminum single crystals, the striated substructure increased proportionately to the rate of growth. The number of subgrains increased from 13 grains/in^2 in the 1/4-inch per hour section (a 1-inch length) to 27 grains/in^2 in the 1-inch per hour section (also a 1-inch length).

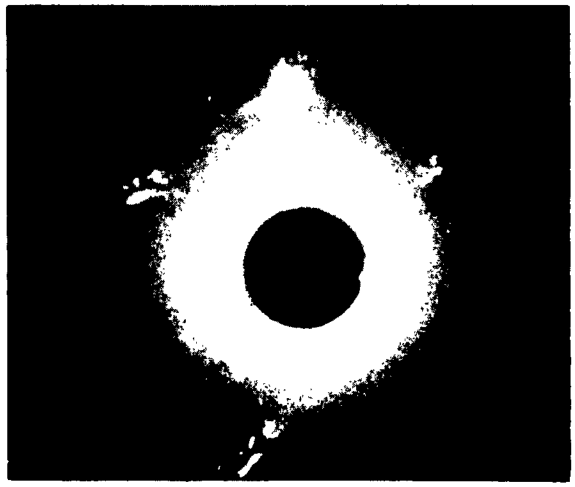
Laue back-reflection x-ray photographs were taken at points along the single crystal length corresponding to the 1/4-inch per hour, 1/2-inch per hour, 1-inch per hour sections and the 1/2-inch per seed section. These photographs reveal split Laue spots indicating subgrain misorientations of about 1 degree in the 1/4-inch per hour and 1/2-inch per hour sections and 1/2 to 1 degree in the 1-inch per hour section. (See Figure 3a, 3b) There was more splitting of spots in the 1-inch per hour section which correlates with the greater number of subgrains evidenced in this section. X-ray photographs were again taken at similar positions along the crystal length (with the exclusion of the 1-inch per hour section) after vacuum annealing* (5×10^{-7} mm Hg) at 1050°C for 100 hours. The absence of splitting in Laue spots and the uniformity of the diffraction patterns at each position of the crystal confirms the increase in perfection due to annealing. (See Figure 3c) A visual examination of the etched structure also confirmed the absence of striated substructure. During vacuum annealing some recrystallization occurred, which may be attributable to deformation imparted in handling the specimen. X-rays were not taken in the recrystallized grains.

Two types of spectrographically analyzed copper were purchased from the American Smelting and Refining Co. 99.000+ purity in the form of a 3/8-inch diameter coil, and 99.99+ purity in the form of 1/4-inch x 12-inch x 4-inch strips. Using the crystal growing equipment, the copper is being vacuum melted at 10^{-5} mm Hg into cylinders to facilitate prep ration of subsequent single crystals. Three single crystal copper seeds of 99.99+ purity with growth axis within 2 degrees of direction [111], [110], and [100] have been obtained from Metals Research Limited, England. These seeds

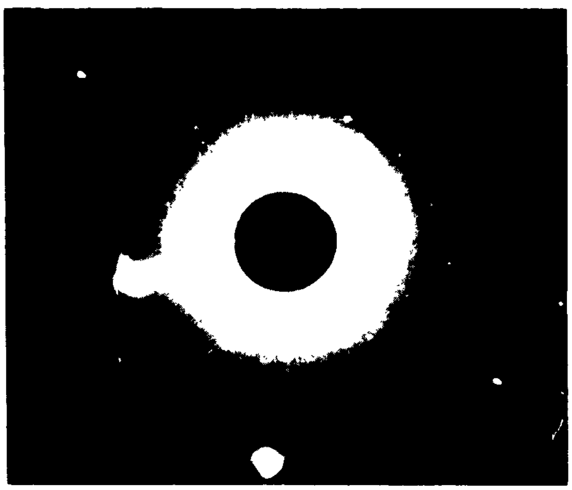
* A special inconel retort was constructed and calibrated prior to vacuum annealing the single crystal.



a) X-Ray of Section Grown at 1/2-Inch Per Hour



b) X-Ray of Section Grown at 1-Inch Per Hour



c) X-Ray After Vacuum Annealing At 1050°C For 100 Hours

Figure 3. Laue Back-Reflection X-Ray Photographs of Copper Single Crystal

will be etched down to predetermined dimensions to allow for expansion during melting in the graphite molds. The diameter of the copper single crystals was determined by the size of the quartz transducers used for excitation: 0.5-inch diameter. The length of the single crystal specimens was based on the design of the tension-compression grips as well as the extensometer to be used. A copper specimen of 0.617-inch diameter by 2.8-inches long was selected for testing.

B. ELECTROCHEMICAL CUTTING AND CHEMICAL POLISHING OF COPPER

There is evidence that the deformation imparted to single crystals during cutting and polishing is not readily removed by etching. F.W. Young[8, 9] has shown that dislocation motion will occur in single crystals of 99.999% copper at stresses as low as $4 gm/mm^2$ (5.7 psi). It was decided to use an electrochemical cutting process which would impart the least amount of deformation to the specimen.

Based on the work of Avery et al.[10], an electrochemical cutting machine was fabricated. As indicated in Figure 4, a revolving disc immersed in a tray of acid establishes part of an electrolytic cell with the specimen and disc being the anodes, and the copper sheet the cathode. The specimen is set a few millimeters from the disc and as the latter revolves, acid fills the gap completing the circuit. While the copper is electrolytically removed, being first plated onto the disc and then replated onto the copper sheet, the counter-weight is moved inward to lower the specimen, thus, maintaining the gap. Several specimens were successfully cut using this machine, once the power conditions were established. A more sophisticated machine was designed and fabricated which features an automatic specimen lowering mechanism, a double disc arrangement, and an interchangeable chemical polishing assembly to provide accurate cutting and polishing (See Figure 5).

The procedure outlined by Young and Wilson[11] was used to polish flat ends on the single crystals of copper after cutting. A small motor-driven chemical polisher was devised in which the copper specimen was supported

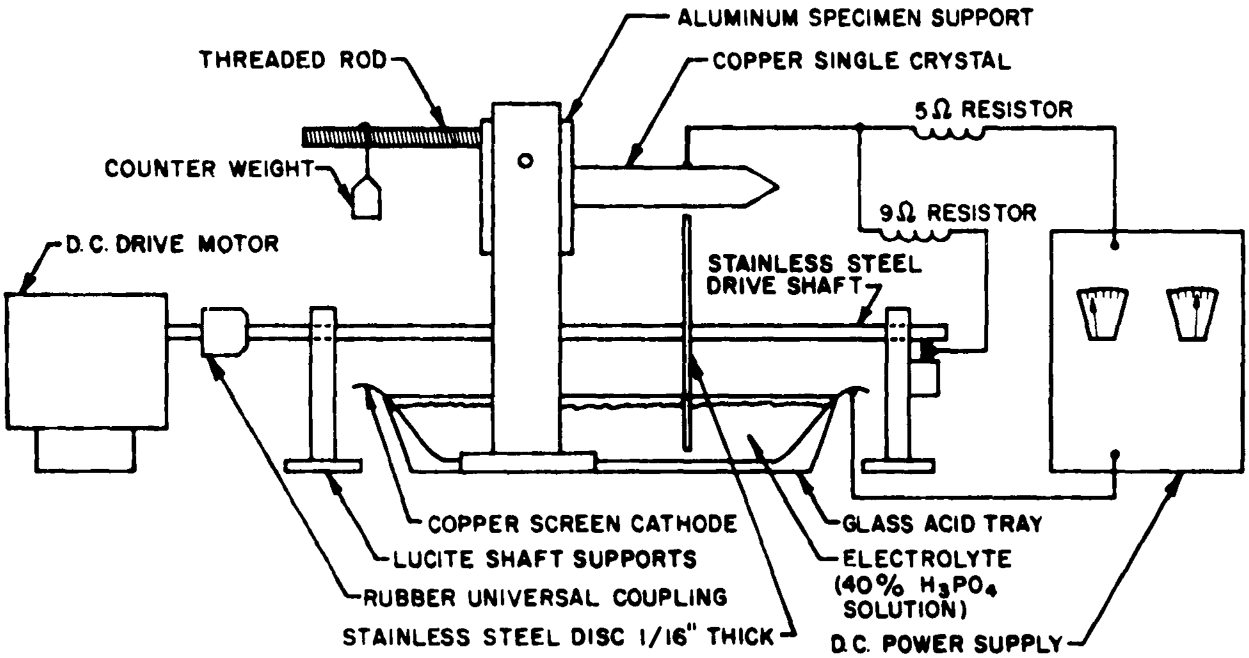


FIGURE 4. ELECTRO-CUTTING APPARATUS

on a movable block, as indicated in Figure 6. The specimen was moved into contact with the cotton-cloth covered disc which revolved through an acid bath*.

All single crystal specimens will be cut and polished to within 50 millionths of an inch. This degree of parallelism is necessary to ensure proper reflection of the ultrasonic waves to be propagated through the specimen. Parallelism will be measured with a Federal Gage sensitive to within 5 millionths of an inch.

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* A saturated solution of $CuCl_2$ in HCl.

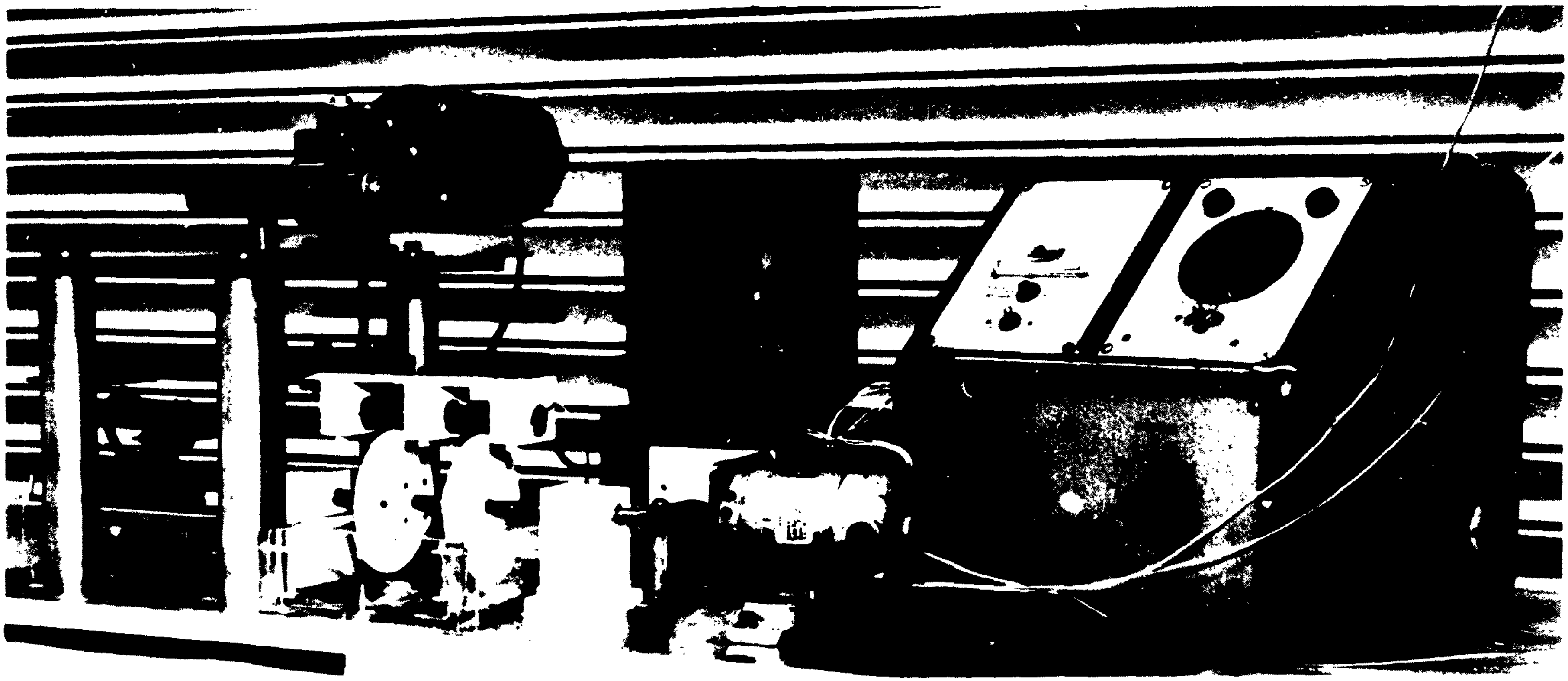

Figure 5. Machine for Electrochemical Cutting and Polishing of Copper Single Crystals

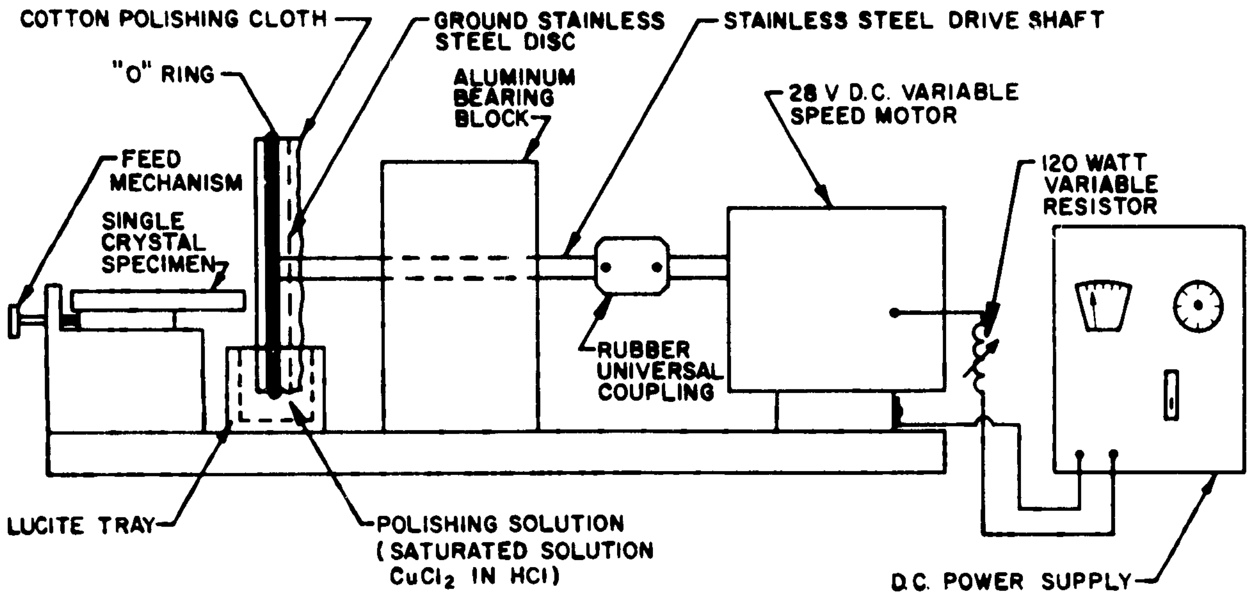

FIGURE 6. CHEMICAL POLISHER

C. TESTING OF COPPER SINGLE CRYSTALS IN TENSION AND COMPRESSION

The arrangement of the fixtures in the testing of copper single crystals in tension and compression is shown in Figure 7. A Baldwin 50,000 lb test machine will be used, equipped with a highly sensitive 500 lb load cell with 10, 25, 100, and 500 lb ranges. Self-aligning grips were designed and fabricated to transmit the tension and compression loads to the single crystal specimen through ground hemispherical housings (see Figure 8). The single crystal specimen adhesively bonded * to specially machined bronze balls will be placed into the split grips and seated loosely in the hemispherical housings. Both the upper and lower housings will be clamped to a flat steel plate to prevent deformation of the crystal in the grips. The outer sections of the grips will then be joined to the housings. Once completely assembled, the tension-compression grips with the specimen will be attached to the test machine. Calibrated strain gage extensometers will be attached to the specimen as shown in Figure 9.

* Insulweld 213 B&A adhesive

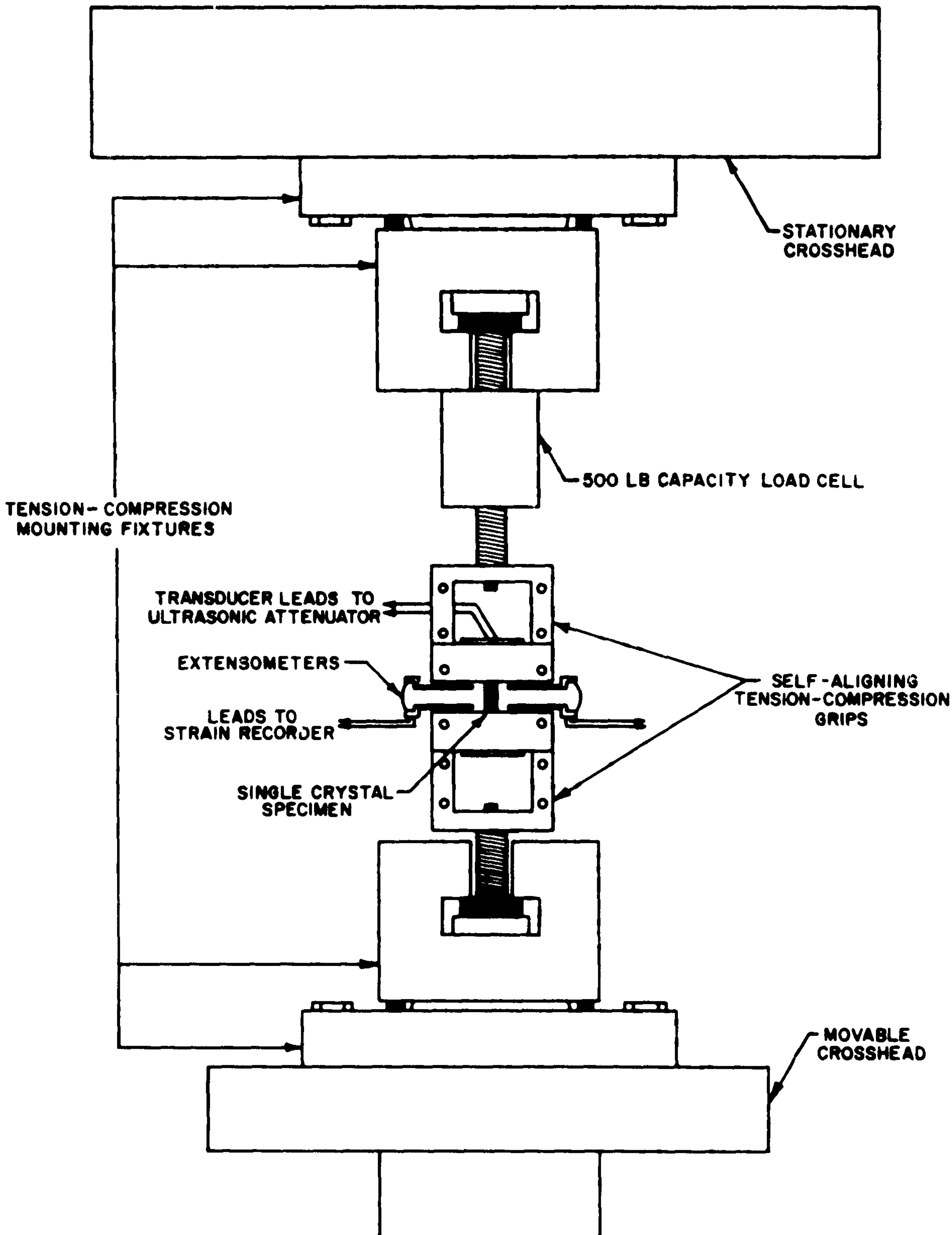

FIGURE 7. TENSION-COMPRESSION TEST FIXTURE

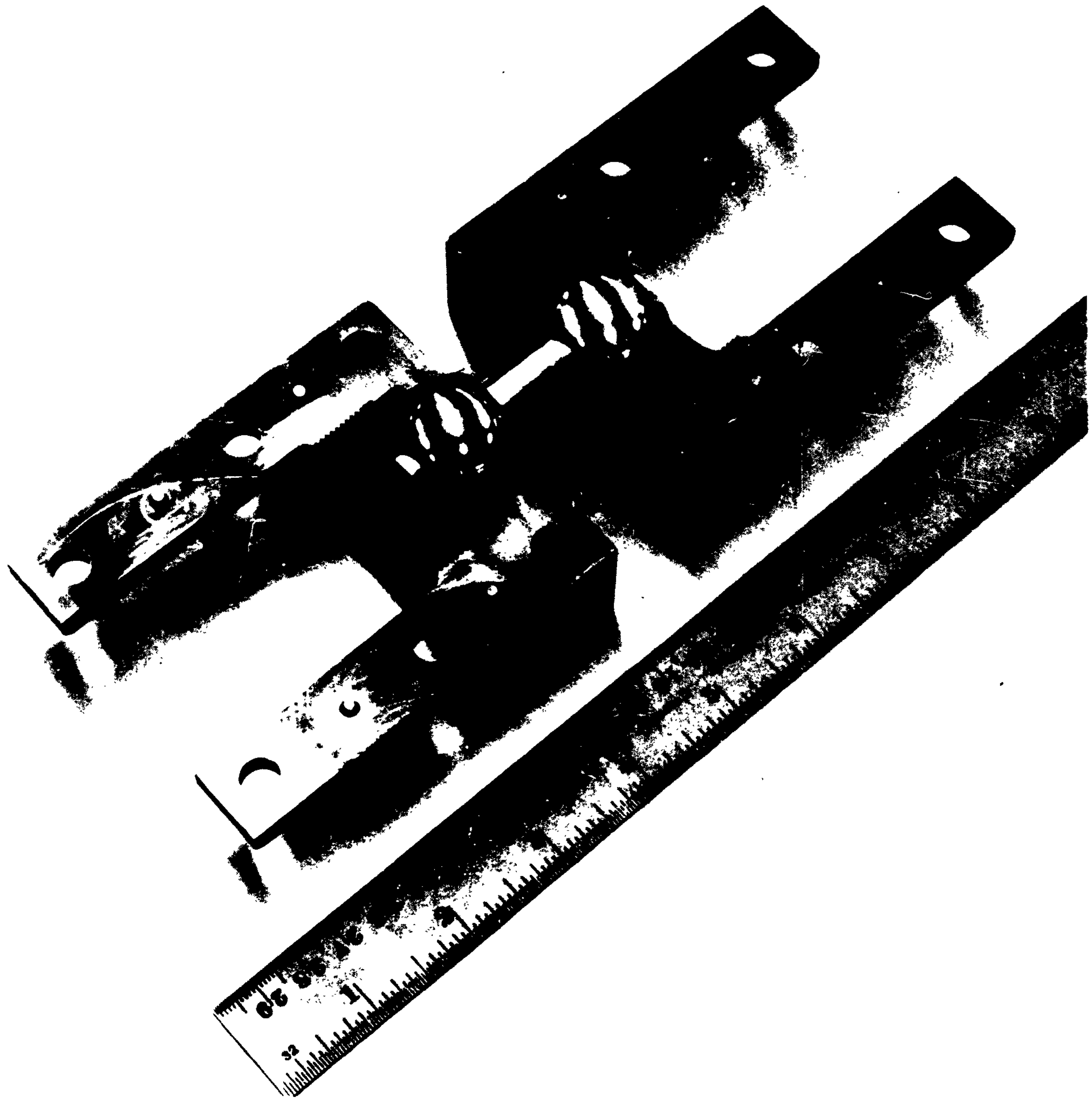

Figure 8. Sections of Tension-Compression Grips Showing Specimen in Ground Hemispherical Housings

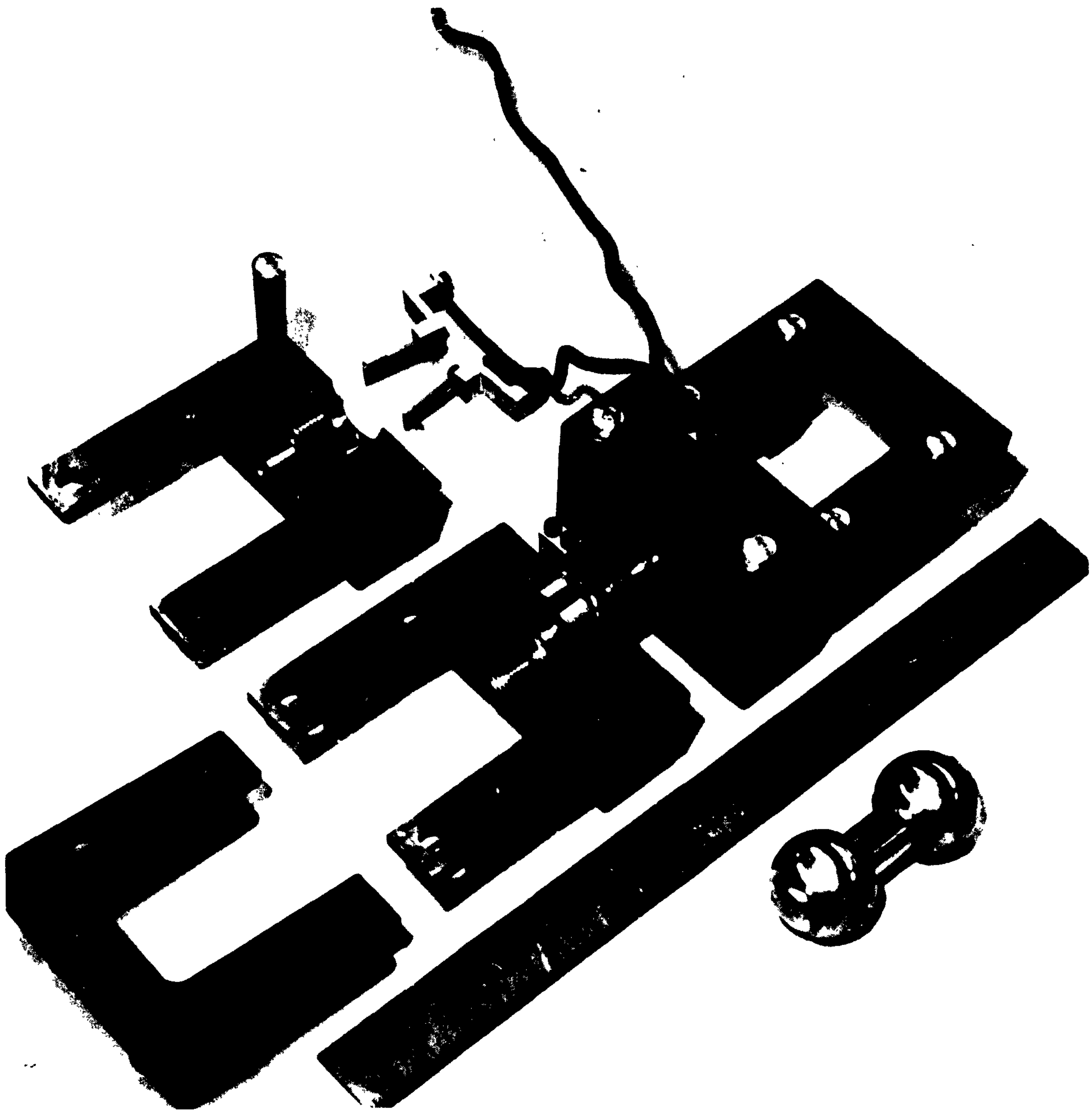

Figure 9. Self-Aligning Tension-Compression Grips for Testing Single Crystals

SECTION III

PROJECTED WORK FOR NEXT QUARTER

- Oriented single crystal specimens of high purity copper will be grown from oriented seeds.
- The power conditions will be established for the electrochemical cutting and polishing machine.
- Calibration tests will be performed on polycrystalline specimens to determine the eccentricity of loading in tension and compression.
- The reproducibility of testing, and the strength of the adhesive bonds will be determined in tension and compression.
- Comparisons in ultrasonic attenuation will be made between as-grown and vacuum annealed specimens to determine whether subsequent specimens will be vacuum annealed.
- Attenuation measurements will be made on oriented single crystals during cyclic deformation.

SECTION IV

REFERENCES

1. N. Thompson and N.J. Wadsworth, "Metal Fatigue," Advances in Physics 7, 72 (1958).

2. W.A. Wood, "Some Basic Studies of Fatigue in Metals," Fracture Symposium, John Wiley & Sons, Inc., New York (1959).

3. Basic Mechanisms of Fatigue, ASTM Special Tech. Publ. No. 237, June 1958.

4. J.C. Grosskreutz and F.R. Rollins, Research on the Mechanisms of Fatigue, WADC Tech. Rpt. 59-192, Sept. 1959.

5. J.C. Grosskreutz, Research on the Mechanisms of Fatigue, WADD Tech. Rpt. 60-313, April 1960.

6. A. Hikata, R. Truell, B. Chick, K. Lucke J. Appl. Phys. 27, 396 (1956).

7. J.T. McGrath, C.B. Craig, Trans. AIME, 215, 1022 (1959).

8. F.W. Young, J. Appl. Phys. 32, 1815 (1961).

9. F.W. Young, J. Appl. Phys. 33, 3553 (1962).

10. D.H. Avery, M.L. Ebner, W.A. Backofen, Trans. AIME, 212, 256 (1958).

11. F.W. Young, T.R. Wilson, Rev. Sci. Inst. 32, 559 (1961).